3-426 (Revised 1-52)

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OAK RIDGE NATIONAL LABORATORY
Operated By
CARBIDE AND CARBON CHEMICALS COMPANY

UCC

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DATE: July 10, 1953

SUBJECT: METAL RECOVERY PROGRESS REPORT FOR MONTH OF JUNE 1953

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TO: F. L. Culler

FROM: W. H. Lewis, J. L. Matherne, R. E. Brooksbank

CLASSIFICATION CANCELLED
DATE 8-14-62
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[redacted] COVER SHEET
[redacted] INFORMATION

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[redacted]

1.0 SUMMARY

During June 8000 kg of uranium was recovered from W-10 metal wastes. The solvent extraction losses for uranium were 0.04 per cent. The composite uranium loss for the plant, which includes losses resulting from centrifuge cleanouts and product filtration operation plus solvent extraction losses, was 1.9 per cent. The fission product beta and gamma activities of the recovered uranium (after one cycle process) were 2.1 per cent and 37 per cent, respectively, of natural uranium. The plutonium contamination in the uranium shipped during the month was 10 ppb.

No plutonium was recovered from the metal waste solutions during June. A leak, caused by corrosion, developed in the steam coil of the IBP evaporator in late May. Inspection of the evaporator to determine the cause of failure revealed that part of the coil was made of unstabilized 309 stainless steel.

The plant had an on-stream operating time of 28 days, with an average uranium output of 283 kg (624 lb) per operating day. The column downtime of three days was due to failure of the solution removal pump on the product evaporator.

Approximately 1500 kg of uranium was recovered that did not meet the specification for plutonium content. By feeding the rejected uranium into the plant when the feed preparation was limiting the plant capacity, all the rejected material was processed at no extra cost.

Three shipments of uranyl nitrate solution containing 6800 kg of uranium were transferred to the Mallinckrodt Chemical Company for denitration. A total of 26,000 kg of uranium has been shipped to date.

The capacity of the Metal Recovery plant continues to be limited by the concentration of the slurry from the metal waste tank W-10. The average uranium concentration in the slurry pumped from waste tank W-10 during the month was 36 g/liter.

Nitric acid recovery averaged approximately 24 per cent for the month. The high rate of corrosion in the nitric acid recovery equipment is caused by the presence of fluorides and chlorides in the W-10 waste solution. Corrosion test plates placed in the reboiler section of the nitric acid still indicated a local corrosion rate of 150 mils/yr. In late June, the steam coil in the nitric acid reboiler developed a leak. This is the second reboiler steam coil that has failed as a result of corrosion in the last six months.

Nitric acid recovery will be discontinued until sufficient data on corrosion control are available.

Because of the difficulty experienced in recovering nitric acid, the quantitative recovery of plutonium is not economically feasible. For this reason, a low-acid flowsheet, which recovers only 80% of the plutonium, will be used. Even under these conditions, the economic feasibility of the plutonium recovery is uncertain.

2.0 PROCESS CHEMISTRY

2.1 Equilibrium Solvent Extraction Losses

The uranium and plutonium losses through one cycle of operation are presented in Table 2.1-1.

Table 2.1-1

Uranium and Plutonium Losses through One Cycle

Material	Flowing Stream Losses (%)					Composite[a] Losses (%)
	IAW	IBP	ICU	ICW	Total	
Uranium	<0.01	<0.01	---	0.04	0.04	1.9
Plutonium	8.5[b]	---	0.50	0.20	9.30	---

(a) Composite losses include all losses from the plant. All waste streams flow to a common catch tank.

(b) The second plutonium cycle was inoperative during the month.

2.2 Decontamination

Through one cycle of operation, the fission product beta and gamma activities were separated by factors of 1.0×10^4 and 1.2×10^3, respectively.

[redacted]

Specific fission product decontamination factors of the uranium product were as follows:

Table 2.2-1

Specific Decontamination Factors for ICU Concentrate

Gross β	732
Gross β (corrected)[a]	1.0×10^4
Gross γ	730
Gross γ (corrected)[a]	1.2×10^3
Ru β	8.0×10^3
Zr β	163
Nb β	801
TRE β	1.0×10^5

(a) Corrected for UX_1 + UX_2 activity.

2.3 Uranium Product Purity

The uranium product, which had a uranium concentration of 442 g/liter, was contaminated with plutonium to the extent of 7 to 14 ppb. The fission product beta and gamma activities were 2.1 and 37 per cent, respectively, of old natural uranium.

Ionic contaminant analysis of the shipment composite sample has been delayed because of inoperative equipment.

2.4 Chemical Flowsheet

During the month of June, a low-acid flowsheet was demonstrated for uranium recovery. It is necessary to use a feed containing 4 or 5 moles of nitric acid per liter (high-acid flowsheet) to recover plutonium quantitatively from metal wastes. Because of the failure of the IBP evaporator, necessitating the shutdown of the second plutonium cycle, a feed containing an average of 2.8 moles of nitric acid per liter (low-acid flowsheet) was used during the month. The plutonium losses in the IA column raffinate were 8 to 11 per cent for the low-acid flowsheet conditions.

[redacted]

[redacted]

A 30 per cent TBP-70 per cent Amsco solvent was used during the month. The relative volumes and compositions of the various streams are given in Tables 2.4-1 and 2.

Table 2.4-1

Stream Compositions and Relative Volumes

Stream	Composition	Relative Volume (IAF=100)
Entering:		
IAF	0.43 $\underline{M}$ U, 2.8 $\underline{M}$ HNO_3	100
IAS	3.0 $\underline{M}$ HNO_3.	25
IAX	28.6% TBP in Amsco	150
IBS	28.6% TBP in Amsco	34
IBX	0.04 $\underline{M}$ $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$, 0.08 $\underline{M}$ H_2SO_3H	25
ICX	Distilled water	240
Exit:		
IAW		125
ICU		240
ICW		184

Table 2.4-2

Flowing Stream Compositions (June, 1953)

Stream	U (g/liter)	HNO_3 ($\underline{M}$)	α (c/m/ml)	β (c/m/ml)	γ (mv/ml)
IAF	101	2.8	3.5×10^4	1.5×10^5	250
IAW	0.0004	2.49	2.4×10^3	---	---
IBP	0.0004	1.75	---	---	---
ICU	37.7	0.06	76	111	---
ICW	0.027	---	50	50	---

[redacted]

The following flowsheet modifications were made during the month:

(a) Emulsification due to poor phase separation and solvent raffinate uranium losses of 0.3 to 0.5 per cent have been noticed in the IC (uranium strip) column. The use of 0.05 M HNO_3 as the IC column strip gave good phase separation and reduced the raffinate loss by a factor of 9.

(b) The original metal recovery flowsheet made use of a 0.005 M HF solution as the IB column plutonium partitioning agent because of its nonsalting properties during evaporation. Prior to the evaporation of the plutonium-bearing aqueous stream from the IB column, sufficient $Al(NO_3)_3 \cdot 9H_2O$ (0.05 M Al) was added to complex the fluoride, thereby eliminating corrosion. Further investigation indicated that the aluminum would not satisfactorily complex the fluoride, and corrosion difficulties persisted in the IBP evaporator. As a substitute for the HF, ferrous sulfamate was used in the IBX stream.

Preliminary indications of a badly corroded IBP evaporator, necessitating the shutdown of the second plutonium cycle, offered an opportunity to investigate the feasibility of using 0.005 M HF in the IBX stream. Utilization of HF as a plutonium partitioning agent in the IB column (assuming that no plutonium would be recovered) would reduce the chemical consumption cost as well as the iron contamination (292 ppm) in the uranium product caused by the entrainment of ferrous ammonium sulfate. During the 16-hr test period, analysis of the concentrated uranium product indicated that the plutonium contamination was 100 parts per billion parts of uranium, greater than specifications by a factor of 10. During this test period 250 kg of off-specification material was produced.

[redacted]

3.0 PLANT PERFORMANCE

3.1 Feed Preparation

3.11 Slurry Dissolution

There was 52,140 gal of metal waste slurry pumped from W-10 waste tank to the Metal Recovery plant for feed makeup. A volume of 7400 gal of concentrated nitric acid was added to the slurry for neutralization and uranium dissolution. The average analysis of the dissolved slurry was as follows:

Uranium	35.9 g/liter
Nitric Acid	1.25 M
Specific Gravity	1.12

3.12 Feed Evaporation

There was 59,542 gal of dilute feed concentrated in the feed evaporator to obtain 25,503 gal of concentrated feed containing 110 g of uranium per liter and 2.9 moles of HNO_3 per liter.

During the month 948 gal of unacceptable feed was returned to W-10 tank because of excessive solid precipitation after boildown. This precipitation was caused by an unusually low acid concentration (0.9 M) in the dissolved slurry before evaporation. Laboratory investigation of the solids is now in progress.

3.13 Centrifugation

The evaporated feed flows by gravity to a 26-in. Bird centrifuge at a nominal rate of 170 gal/hr. At this flow rate the solution being centrifuged has a residence time of about 7 min in the centrifuge. This time is sufficient to give a clear product.

3.14 Feed Preparation Time Cycle

The average time required to prepare feed from an 1800-gal batch of slurry was 20 hr. Based on the current uranium concentration of 35 g/liter,

[redacted]

the 1800 gal of slurry requires the following time distribution for preparation:

Item	Time (hr)
Load and heat evaporator	2.0
Evaporation (130 gal/hr)	9.5
Cooling time (to $60^{\circ}C$)	3.0
Centrifugation[a] (175 gal/hr)	3.6
Centrifuge cleanout	2.0
Total	20.1

(a) Includes time required for centrifuging 10 per cent jet dilution.

3.2 Solvent Extraction

During the month a modified Purex flowsheet was used as described in Sect. 2.4.

The following solvent extraction difficulties were encountered during the month:

(a) There was 1553 kg of uranium product which failed to meet plutonium specifications (< 10 parts of plutonium per billion parts of uranium). The IA column pulser amplitude adjustment was inadvertently set to give an amplitude greater than 1 in. This increased pulse amplitude caused considerable quantities of acidic aqueous material to be entrained to the IB column, resulting in ineffective U-Pu partitioning owing to excessive acid concentration in the IB column. The off-specification material, along with the rejected 250 kg from the HF test run (see Sect. 2.4) was processed during periods when plant production was limited by the uranium concentration of the W-10 slurry.

(b) Momentary uranium losses of 2.0 per cent in the IAW stream have been observed from time to time. Considerable difficulty in metering the solvent to the columns has been encountered because of crud formation on rotameter floats and walls. During these temporary column upsets, insufficient solvent was being delivered to the columns as indicated by analysis of the IAP stream. This condition is being controlled by visual observation of the IAW and IBP streams and the adjustment of solvent flow rates. During the next scheduled shutdown of the system, the inventory of solvent will be replaced by 400 gal of pretreated solvent (HNO_3, $Ca(OH)_2$, silica gel), and rotameters will be cleaned.

3.3 Plutonium Second Cycle

Shutdown of the IBP evaporator and the second plutonium cycle was necessary because of a leak which had developed in the steam coil. Visual inspection of the corroded steam coil indicated localized corrosion, suggesting that the coil was partially 309 stainless steel rather than the 309 Scb as specified. Specimens of both sections of the coil have been submitted for chemical analysis to verify this assumption.

3.4 Acid Recovery

The nitric acid vapors evolved during the concentration of the IA column raffinate were concentrated in a bubble type acid still. The average concentration of the recovered acid was 9.8 M and of the waste vapors from the acid still was 0.08 M, and nitric acid recovery averaged 23.7 per cent. Nitric acid recovery is discussed in Sect. 6.0.

During the month, 7117 gal of 13.7 M HNO_3 was transferred into the plant. Nitric acid consumption was 11 moles per mole of uranium produced.

3.5 Solvent Recovery

The solvent raffinate stream from the IC column was contacted with 0.1 M Na_2CO_3 solution in an 8-in. pulse column and then water washed in a 5-in. unpacked glass column.

3.51 Losses

There was 1764 gal of solvent pumped to the columns per day, with an average loss of 3.1 gal per operating day. The average solvent loss for the month was 0.2 per cent of the total solvent throughput.

3.52 Decontamination

Continuous scrubbing of the solvent raffinate streams with 0.1 M Na_2CO_3 effected a uranium decontamination factor of 3. Analysis of the treated solvent stream (ICW) was as follows:

	ICW	Recovered Solvent
Uranium (g/liter)	0.03	0.01
Gross β (c/m/ml)	<50	< 50
Gross γ (c/m/ml)	<50	< 50

4.0 SUMMARY OF PLANT OPERATING COST FOR MAY*

4.1 Gross Cost

The gross operating cost for the Metal Recovery Plant for May was $37,323. The component costs were:

Labor	$14,932
Material	6,547
Overhead**	15,844
Total	$37,323

* Because of the lag in the receipt of cost information, this section of the monthly report covers data for May rather than for the current month.

** The item "overhead" includes charges for expense allocation, the Laboratory Research Director's Division, and the Chemical Technology Division Director.

4.2 Labor Cost Breakdown

The labor charges for May were distributed as follows:

Pilot Plant Section	$8,274
Engineering and Maintenance Division	2,689
Analytical Chemistry Division	3,800

The Analytical Chemistry Division performed 1708 analyses for the plant during May. The average cost per analyses was about $3.90, including labor, material and overhead.

4.3 Operating Cost for Uranium Recovery

The operating cost during May was $2.70 per pound of uranium recovered.

4.4 Summary of Engineering and Maintenance Costs during May

Of the $2689 spent for engineering and maintenance during May, $1080 was spent on equipment and building repairs. This work was covered by blanket work orders. A breakdown of this work by craft follows:

Craft	Man-hours
Pipefitter	270
Millwright	88
Riggers	42
Instrument mechanic	30
Welder	21
Electrician	15
Carpenter	14
Machinist	11
Painter	8
Laborer	2
Total	501

A job description of all blanket work orders is given in the Appendix.

The remaining $1609 was charged to separate work orders as follows:

Description	Amount
Fabrication of stainless steel plutonium resin column	$ 829
Miscellaneous equipment for 2-MR cycle	240
Pulsers for 2-MR cycle	540
Total	$1609

5.0 PERSONNEL RADIATION EXPOSURES

The average radiation exposure of personnel in the plant during the month of June was 116 mr/week.

6.0 OUTSTANDING PROBLEMS

6.1 Feed Preparation

The design capacity of the metal recovery plant is 750 lb/day when a feed containing 100 g of uranium per liter is being processed. The original equipment provided for feed preparation consisted of means for air sparging the metal waste in tank W-10 and a submerged centrifugal pump for pumping the slurry to a tank for neutralization and dissolving in nitric acid. Initial operation in this manner failed to produce a feed having a uranium concentration greater than 33 g/liter, thereby resulting in a plant capacity 33 per cent of design. All attempts to concentrate the slurry by settling were unsuccessful.

[redacted]

During March, steam coils were installed in an existing feed tank for concentration of acidified W-10 slurry to obtain a feed having a uranium concentration of 100 g/liter. The capacity of this evaporator and auxiliary equipment is such that plant design capacity (750 lb of uranium per day) is attained only if the uranium content of the acidified W-10 slurry is greater than 42 g/liter. Figure 6.1-1 presents a correlation of the effect of uranium concentration of the acidified slurry upon the production of the Metal Recovery Plant. This correlation emphasizes the fact that the plant capacity is limited by the uranium content of the slurry below a level of 45 g/liter, while above this level the feed pump capacity is limiting.

Agitation of the metal wastes by recycling the slurry back to W-10 through existing entry ports permitted increasing the uranium content of the acidified slurry to 55 to 60 g/liter. However, during the last few months the concentration has steadily decreased to a June average of 36 g/liter. All attempts to improve this situation with existing equipment have been unsuccessful.

A work order has been submitted to the Engineering Division for the design of facilities for agitation of the metal wastes by recycling them to W-10 through four new 6-in. ports. If this method is not successful in increasing the uranium content of the acidified slurry, it will be necessary to consider more drastic means, such as mechanical agitation of tank W-10.

6.2 Nitric Acid Recovery

The Metal Recovery Plant was designed to recover 70 to 85 per cent of the nitric acid contained in the waste streams. Nitric acid recovery was necessary for the following reasons:

(a) To reduce the chemical cost of uranium recovery.

(b) Quantitative recovery of plutonium from the metal waste requires the use of 1.8 times more nitric acid than is required for recovery of uranium alone. An efficient recovery of nitric acid would permit the recovery of plutonium at no added chemical cost.

6.21 Performance of the Nitric Acid Recovery System

During the past four months of operation the nitric recovery for the plant has averaged 40 per cent. The low acid recovery is due to insufficient evaporating capacity of the IAW evaporator. The overall heat transfer coefficient for the evaporator has been determined to be greater than 400 Btu/ft^2/^{o}F/hr. This heat transfer coefficient indicates satisfactory evaporator operation.

[redacted]

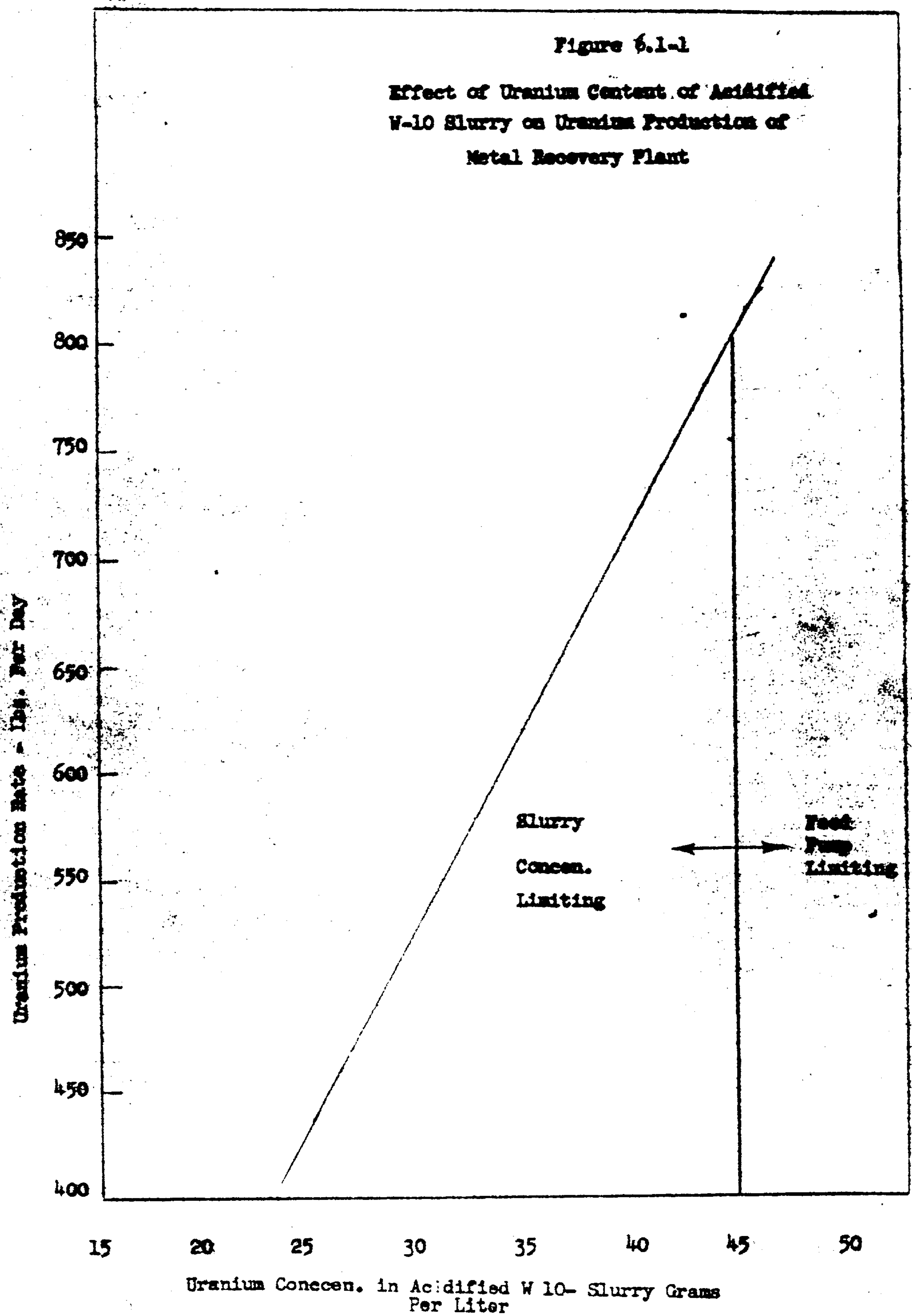
Figure 6.1-1
Effect of Uranium Content of Acidified
W-10 Slurry on Uranium Production of
Metal Recovery Plant
850
800
750
700
650
600
550
500
450
400
Uranium Production Rate - lbs. Per Day
Slurry
Concen.
Limiting
Feed
Pump
Limiting
15
20
25
30
35
40
45
50
Uranium Conecen. in Acidified W 10- Slurry Grams
Per Liter

[redacted]

The IAW evaporator operating under optimum conditions has an evaporation capacity of 50 to 60 gal of water per hour. The flow rate of IAW raffinate to the evaporator is 47.5 gal/hr when the plant is operated at a rated throughput of 750 lb of uranium per day. At this throughput, nitric acid recovery is possible only by a straight evaporation, i.e., there is little excess capacity which would permit the addition of water to the AW evaporator. The amount of nitric acid that can be recovered by straight evaporation depends on how near one is able to approach the "freezing" point of the highly salted concentrate. Operation within several degrees of the freezing point of the concentrate has proved to be hazardous, and is not to be recommended for continuous radioactive processing. Frequent plugging of control instrument lines has necessitated lowering control points to a safe operating range that will permit continuous, trouble-free operation. With safe operating conditions, the best nitric acid recovery obtained to date when operating the plant at a rated uranium throughout of 750 lb/day is about 30 per cent.

Higher nitric acid recovery (approximately 50 per cent) has been obtained when the plant has been operated at a lower uranium throughput. This increase in recovery is due to the addition of extra water to the evaporator pot, thereby effectively changing the solubility equilibrium of the concentrate.

6.22 Corrosion in Nitric Acid Recovery System

Since beginning the Metal Recovery program, excessive corrosion has been observed in the nitric acid recovery equipment. Leaks due to corrosion have developed on the IAW evaporator vapor head and the steam coil and shell of the nitric acid still reboiler. The bubble caps on the two lower plates in the still have become so badly corroded that they have been removed.

The high rate of corrosion in the acid recovery equipment is believed to be caused by fluorides and chlorides contained in W-10 waste solution. The metal feed prepared from W-10 slurry contains 250 ppm of fluorides and 200 ppm of chloride. During the evaporation of the IAW for nitric acid recovery, approximately 30 to 50 per cent of the fluorides in the IAW are distilled over with the nitric acid vapors. The recovered nitric acid from the nitric acid still contained an average of 300 ppm of fluorides.

Unsuccessful attempts have been made to complex the fluoride with aluminum to prevent distillation during evaporation (ORNL CF-52-11-76).

[redacted]

Eight test plates of 347 stainless steel were placed in the reboiler on the nitric acid still for the purpose of determining the local corrosion rate. The results of this test indicate an average corrosion rate of 150 mil/yr. The average exposure time of the plates was 168 hr.

Chemical analysis for chloride in the recovered acid indicated a concentration of 65 ppm. However, it is felt that chlorides are being distilled over in greater quantities than is indicated by the analyses of the recovered acid. The bulk of the chlorides are probably being concentrated in the 110°C temperature zone in the still. This may explain the intense corrosion that occurred on the two lower bubble cap plates. Inspection of the IAW evaporator vapor head reveals considerable pitting, which is characteristic of chloride corrosion.

6.23 Conclusions

(a) The nitric acid recovery system in the plant as originally designed does not have sufficient IAW evaporating capacity to give the desired acid recovery of 70 to 85 per cent. In order to ensure high nitric acid recovery coupled with permissible operating conditions, the IAW evaporator should have an operating capacity three times the present rate. Additional capacity in the IAW evaporator would permit the addition of extra water, which has been shown to increase the recovery of nitric acid from a highly salted solution. (ORNL CF-51-7-192)

(b) The high rate of corrosion in the nitric acid recovery equipment plus a low nitric acid recovery make questionable the advisability of continuing nitric acid recovery. When a feed containing 2.5 moles of nitric acid per liter is being processed, an acid recovery of 30 per cent would effect a saving of only $0.048 in acid cost per pound of uranium processed.

At the present corrosion rate, savings in chemical cost can easily be cancelled by repair and replacement of equipment. In the last six months of operation, the maintenance repair cost for the nitric acid recovery equipment has amounted to approximately $0.036 per pound of uranium processed.

6.3 Plutonium Recovery

The Metal Recovery plant was designed to recover plutonium along with the uranium from the metal waste solution. The prime reason for considering plutonium recovery was that the value of the recovered plutonium would offset a portion of the cost assessed to uranium recovery. This assumes that the recovery of the plutonium does not add any extra cost to uranium recovery.

[redacted]

The metal waste solutions contain appreciable quantities of sulfates, phosphates, fluorides and other aqueous-soluble complexing agents which inhibit plutonium extraction. Quantitative recovery of the plutonium requires the use of 1.8 times more nitric acid than is required for uranium recovery alone. Therefore, to recover the plutonium at no extra chemical cost it becomes imperative to have a nitric acid recovery system giving 100 per cent acid recovery. However, as pointed out in Sect. 6.2, the acid recovery of the Metal Recovery plant is averaging only 30 to 40 per cent. Assuming an acid recovery of 40 per cent and the requirement of 1.8 times more acid for plutonium extraction than for uranium extraction, quantitative recovery of plutonium entails a chemical cost of $60 per gram of plutonium recovered. An additional cost amounting to $35 per gram of plutonium recovered is incurred for analytical service and chemicals required for operation of the plutonium second cycle equipment. The total estimated cost of $95 per gram for the plutonium recovered in the plant does not include the cost for isolation or any maintenance cost required for the operation of the plutonium equipment.

At the present time, the most obvious way to reduce the cost of plutonium recovery is to lower the nitric acid consumption to the amount required for uranium extraction alone. By this means the feed concentration in nitric acid can be lowered from 4.5 moles/liter to 2.5 moles/liter. This change will increase the plutonium loss in the IA column from approximately 1 to approximately 15 per cent. With a low acid flowsheet, the overall plutonium recovery for the plant would probably not exceed 80 per cent.

It is concluded that column extraction conditions should be dictated by uranium recovery. Quantitative recovery (95 to 100 per cent) of plutonium from metal wastes should be discontinued for the following reasons:

(a) Inefficient acid recovery system in the plant causes high chemical cost that must be assessed to plutonium recovery.

(b) The Metal Recovery process by nature of the feed material is physically a dirty chemical operation which does not favor quantitative recovery of tracer amounts of plutonium (approximately 5 g of plutonium per ton of uranium).

(c) Approximately 80 per cent of the plutonium can be recovered as a "salvage" operation when the plant is operated on a low-acid flowsheet.

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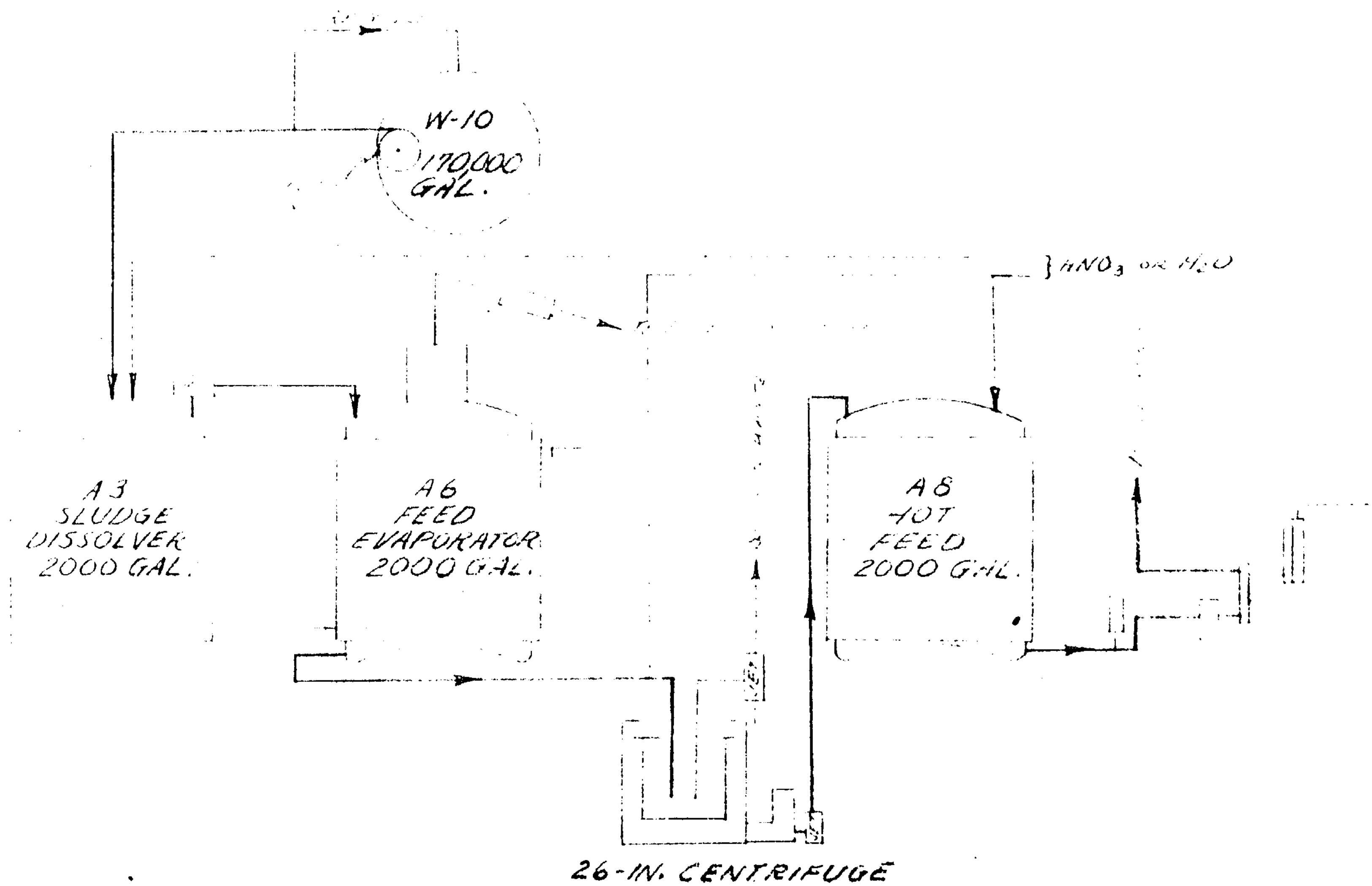
7.0 APPENDIX

[redacted]

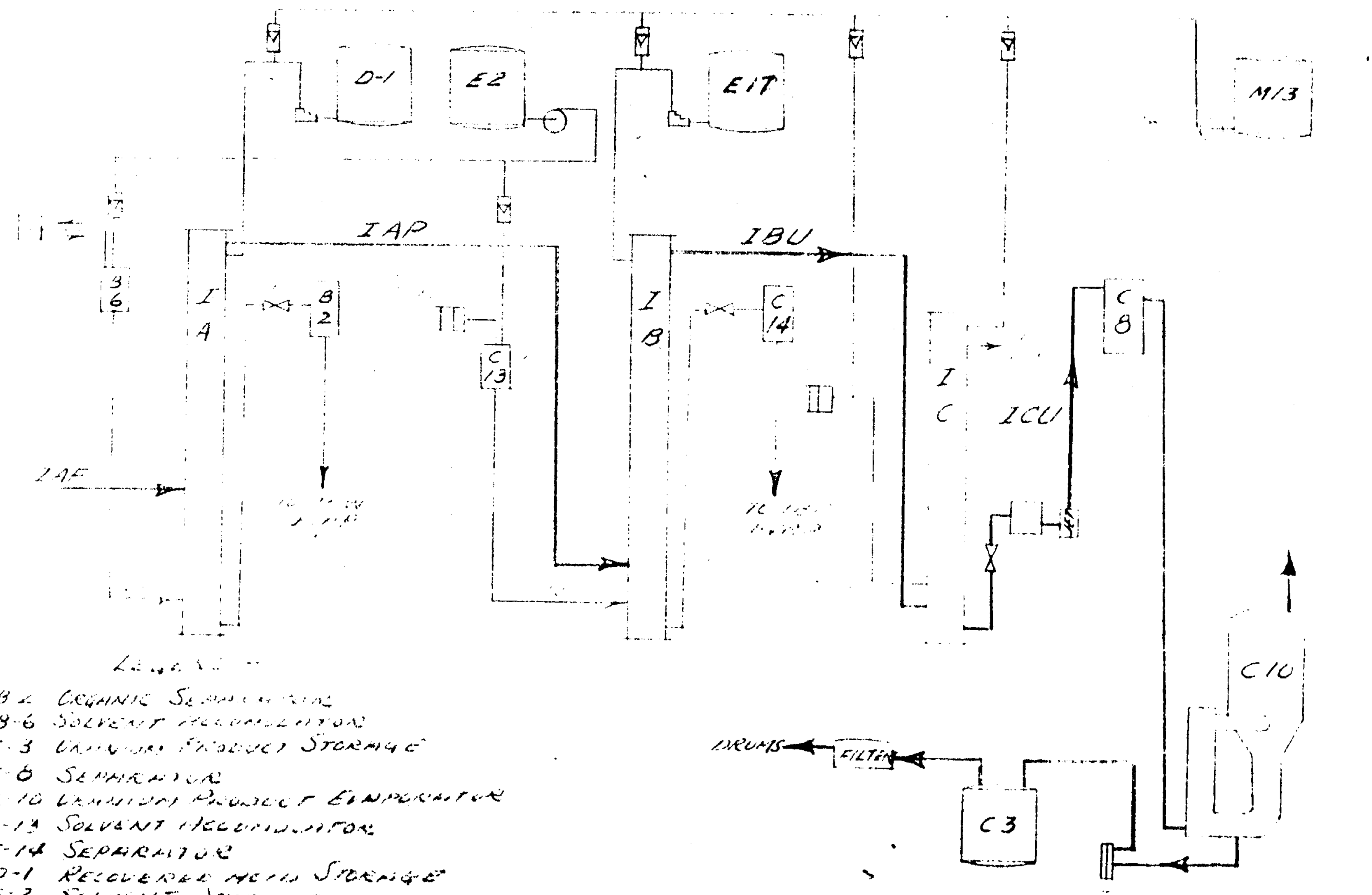
7.1 Equipment Flowsheets

Equipment flowsheets used for the recovery of uranium from tank farm metal wastes are presented as Figs. 7.1-1, 2, 3, 4, and 5.



M.R. EQUIPMENT FLOWSHEET
FEED PREPARATION
FIG. 7.1-1

[redacted]



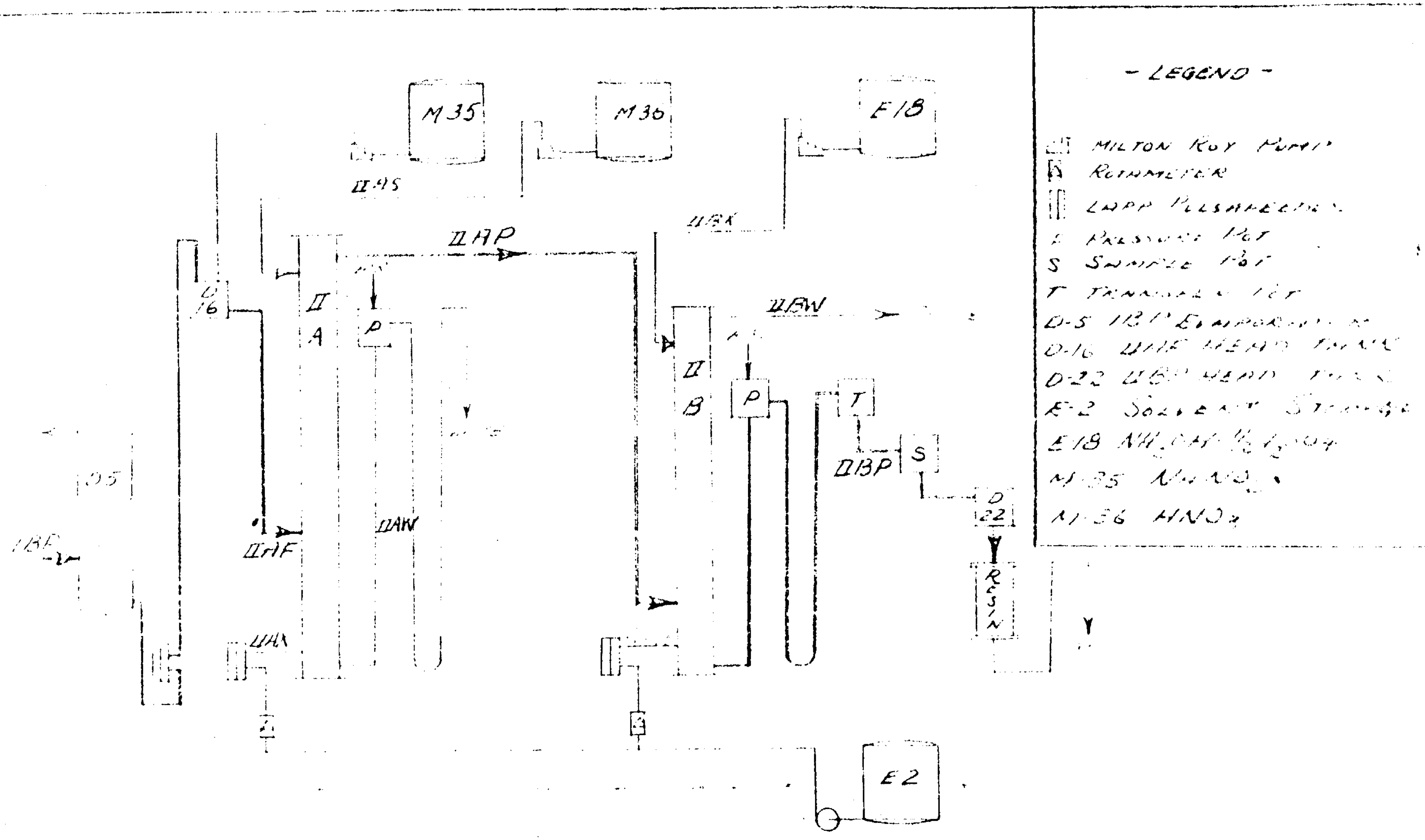
LEGEND

B-2 ORGANIC SEPARATOR
B-6 SOLVENT ACCUMULATOR
C-3 URANIUM PRODUCT STORAGE
C-8 SEPARATOR
C-10 URANIUM PRODUCT EVAPORATOR
C-13 SOLVENT ACCUMULATOR
C-14 SEPARATOR
D-1 RECOVERED METAL STORAGE
E-2 SOLVENT STORAGE
M-13 CONDENSATE STORAGE
E-17 FERROUS AMMONIUM SULPHATE STORAGE

M.R. EQUIPMENT FLOWSHEET
FIRST CYCLE
FIG 9.1-2

[redacted]

Drawing #

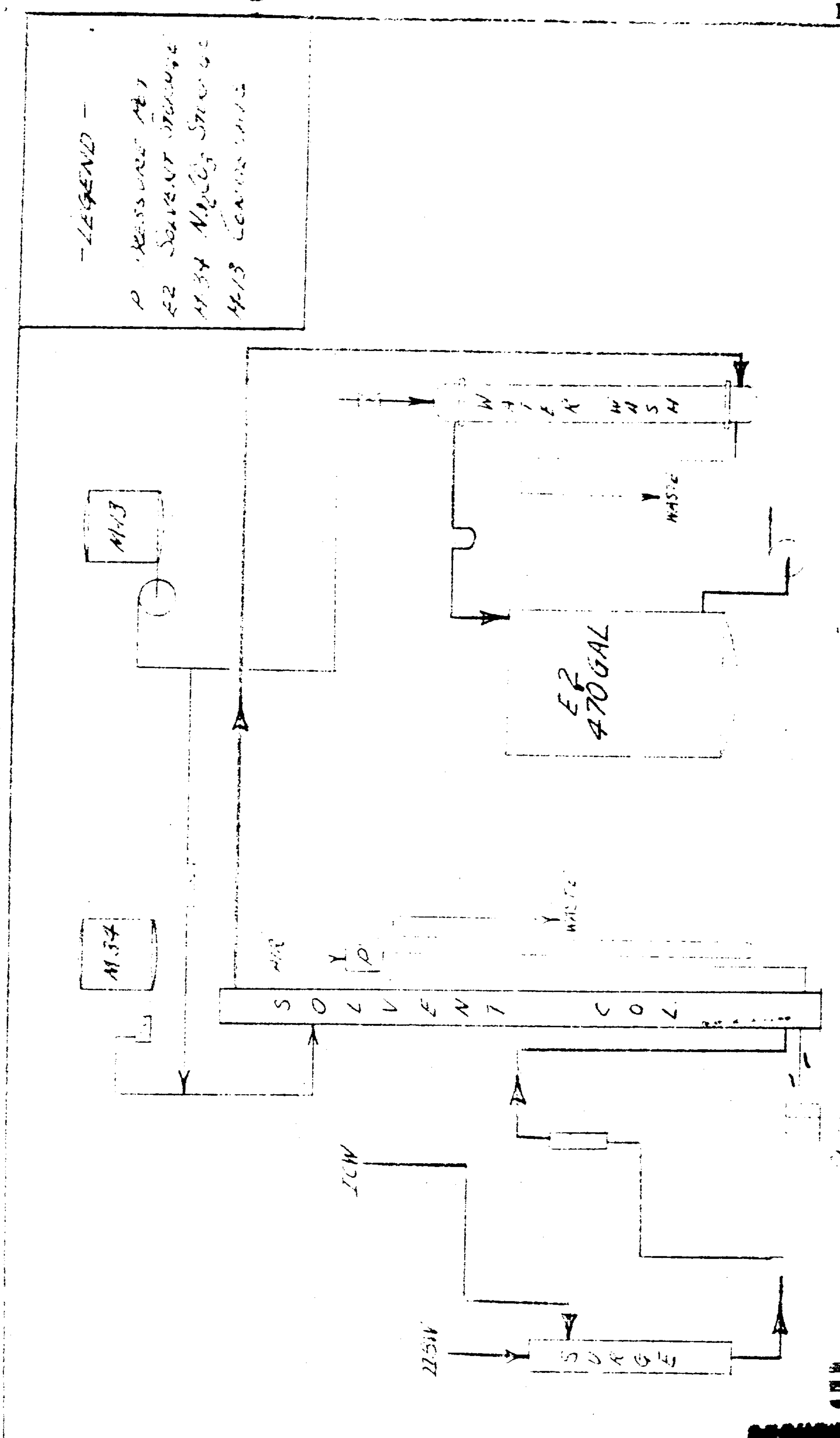


M.R. EQUIPMENT FLOWSHEET
SECOND PLUTONIUM CYCLE
FIG. 7.1-3

[redacted]

SECURITY INFORMATION

Drawing # 20010



M.R. EQUIPMENT FLOWSHEET
SOLVENT RECOVERY
FIG 9.1-4

[redacted]

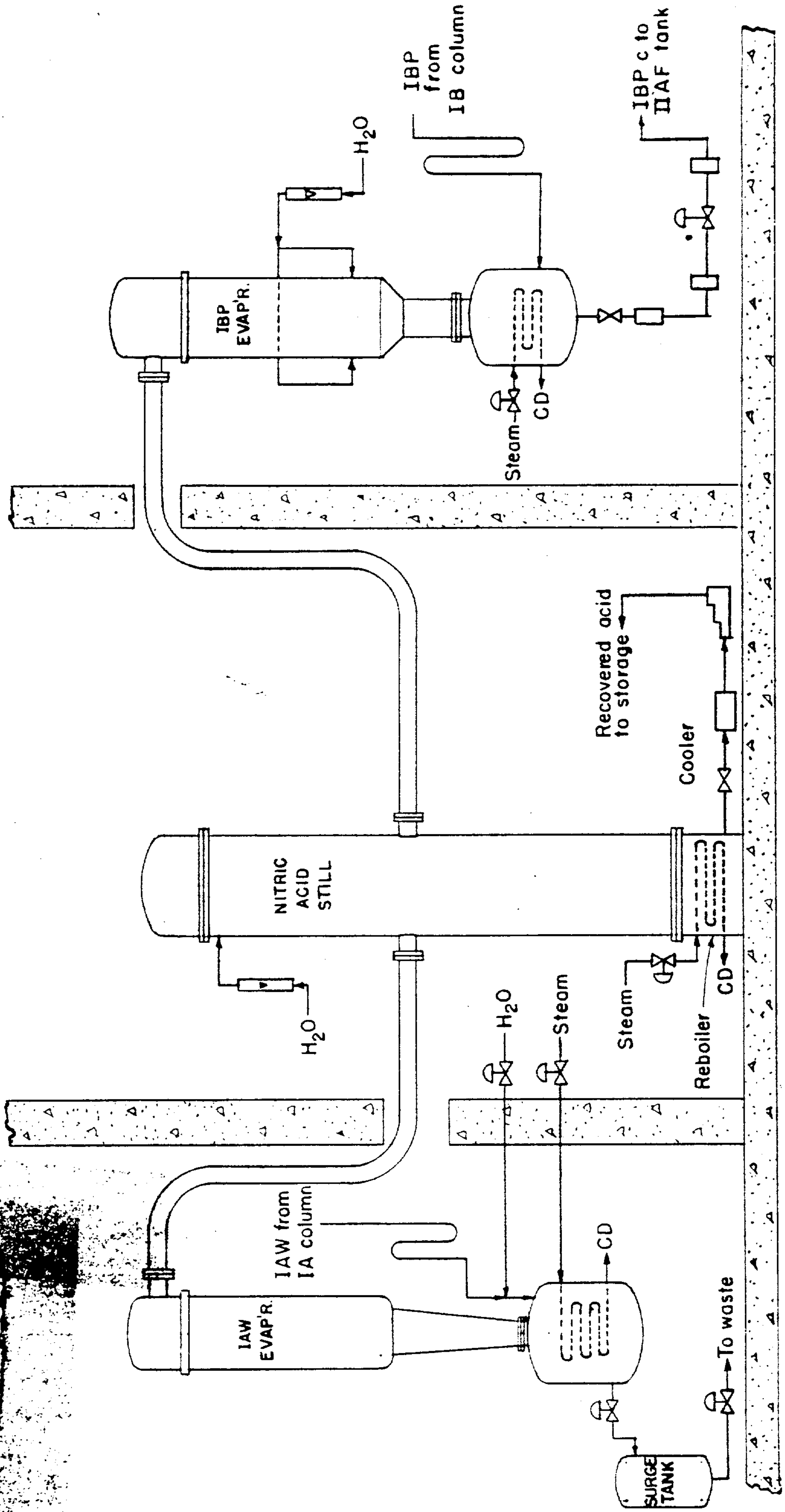


FIG. 7.1-5

ACID RECOVERY SYSTEM METAL RECOVERY PLANT

[redacted]

7.2 List of Blanket Work Orders for May

May Work Order No.	Date	Man Hours Worked	Job Description
1	5-1	14	Repack and replace valve on product filter
2	5-4	17	Repair pulser drive head
3	5-4	2	Move product drums onto skids
4	5-4	8	Miscellaneous maintenance repair in 3505
5	5-4	2	Repair IBP Lapp pump
6	5-5	8	Miscellaneous pipefitting
7	5-5	8	Miscellaneous pipefitting
8	5-6	4	Repair air valve
9	5-6	8	Do necessary machine work on spare pulser
10	5-6	8	Miscellaneous pipefitting
11	5-8	21	Miscellaneous pipefitting
12	5-8	16	Repair IAS pump and repack centrifuge
13	5-11	23	Miscellaneous pipefitting
14	5-11	9	Move product drums onto skids
15	5-11	8	Shore product drums in truck
16	5-11	8	Repair IBP evaporator instruments
17	5-11	3	Do required machine work on air valve
18	5-12	2	Replace light bulbs in cells
19	5-12	18	Miscellaneous pipefitting
20	5-12	4	Disconnect CUC unload pump

[redacted]

[redacted]

May Work Order No.	Date	Man Hours Worked	Job Description
21	5-13	8	Miscellaneous pipefitting
22	5-14	16	Repair acid still thermowell
23	5-15	8	Miscellaneous pipefitting
24	5-16	1	Repair extension cord
25	5-18	8	General painting
26	5-18	8	Disconnect W-10 pump and miscellaneous pipefitting
27	5-19	8	Remove top flange on ICU evaporator Repack IBX-FS pump
28	5-19	6	Oil feed centrifuge
29	5-19	16	Repair leaks in condensate line, IAS acid line, and acid header
30	5-20	6	Check product scales
31	5-20	12	Repair ICU evaporator and install sight glasses in acid still
32	5-20	8	Remove thermowell from ICU evaporator and check IA and IB pulser motor heaters
33	5-20	4	Repair acid still instruments
34	5-20	6	Shore product drums in truck
35	5-20	12	Move product drums to truck and onto skids
36	5-20	9	Remove ICU condenser and vapor header
37	5-21	30	Miscellaneous piping
38	5-21	26	Connect ICU evaporator Condenser and vapor head Repair acid header and acid still steam header
39	5-22	3	Repack acid pump
40	5-22	26	Connect W-10 pump Repair IBX head tank Repair off gas header Install corrosion samples in acid still
41	5-25	4	Check Foxboro D/P cell Check acid meter

[redacted]ON

May Work Order No.	Date	Man Hours Worked	Job Description
42	5-25	8	Remove piping from dissolver pad. Repair leak in acid header
43	5-26	10	Check acid meter
44	5-26	8	Repair acid header and condensate header Remove dissolver pad piping
45	5-27	6	Replace pin in feed Centrifuge skimmer arm
46	5-27,28	16	Replace acid meter Remove piping from dissolver pad, repair acid header
41	5-28	4	Revamp tank for aluminum nitrate storage
48	5-29	8	Replace ICX rotameter Tighten IBX rotameter
49	5-29	12	Move pads from pit
50	5-29	8	Revamp tank for aluminum nitrate storage

[redacted]

Drawing # 20280

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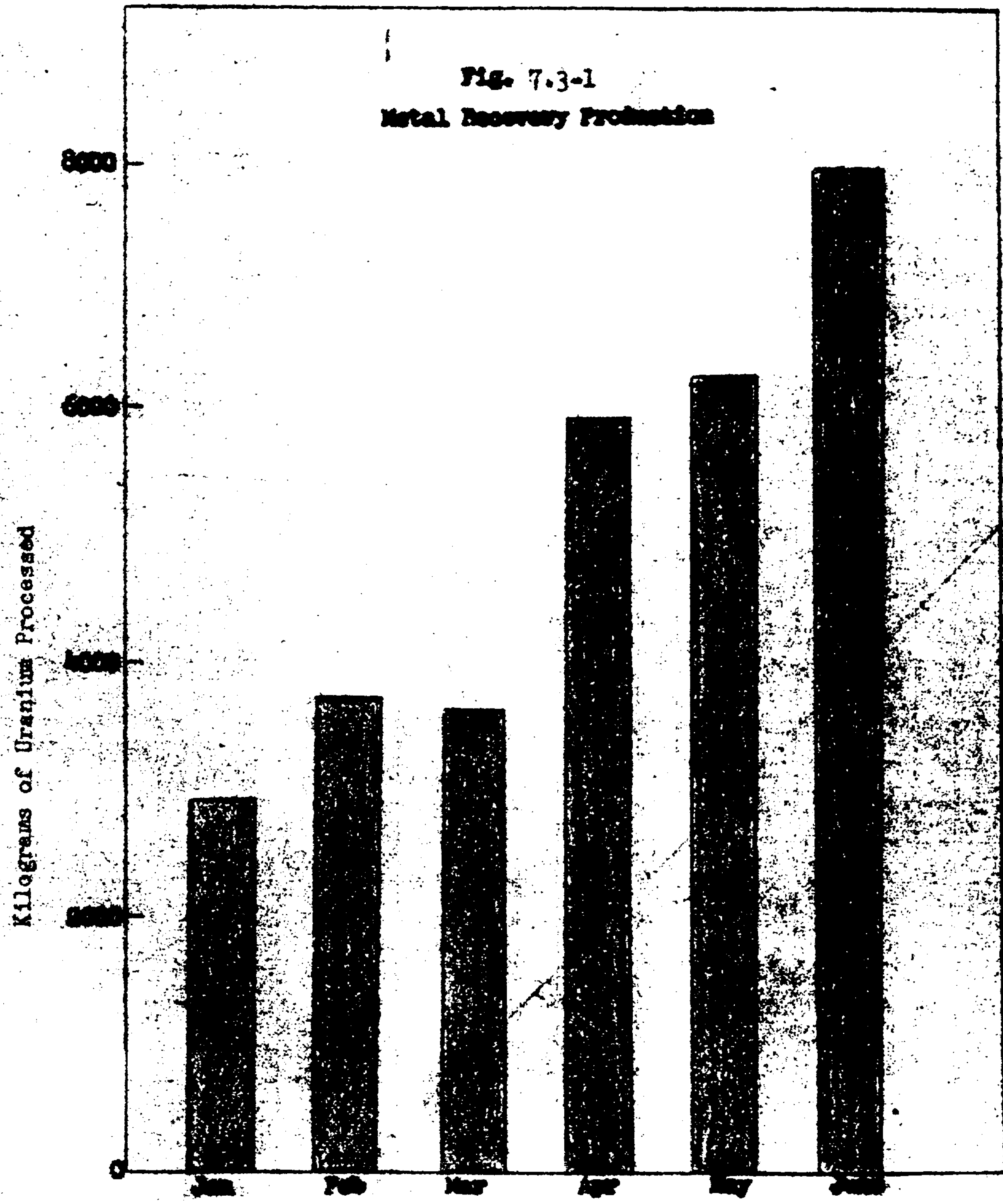

Fig. 7.3-1

Metal Recovery Production

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1785

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X-426 (Revised 1-52)

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OAK RIDGE NATIONAL LABORATORY
Operated By
CARBIDE AND CARBON CHEMICALS COMPANY

UCC

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TO:	F. L. Culler	
FROM:	R. E. Brooksbank	

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CONTENTS

1.0 SUMMARY

During November the Metal Recovery plant operated at 89.9 per cent time efficiency (column on-stream time) and processed an average of 328 kg (721 pounds) of uranium per operating day. A total of 8,846 kgs of uranium and an estimated 42 grams of plutonium were recovered during the month. The 10.1 per cent down time was required to facilitate installation of 2 MR (second uranium cycle) equipment.

The composite uranium loss from the plant, which included all losses incurred in centrifuge cleanouts and solvent extraction, was 1.4 per cent. The uranium loss resulting from solvent extraction was 0.3 per cent.

The fission product beta and gamma activities of the recovered uranium product were 0.5 and 34 per cent, respectively, of natural uranium. The plutonium contamination in the recovered uranium was 27 ppb. The total metallic ion contamination in the uranium was 975 ppm. The main contaminant was iron (900 ppm) and was due to entrainment from the plutonium partitioning column, which will be modified during January to provide a larger de-entrainment section.

During November three shipments of uranyl nitrate product solution containing 6,959 kgs of uranium were transferred to Y-12 plant to be converted to uranium trioxide. A total of 76,866 kg of uranium has been recovered from metal waste solutions up to November 26.

Two shipments of irradiated slugs containing 1,694 kg of uranium were received from Brookhaven National Laboratory during November. Three shipments of waste material in the form of press cake sodium diuranate, containing 5,667 kg of uranium were received from Harshaw Chemical Company.

A graph indicating the monthly uranium production of the plant for the year is presented in Figure 1.0.

Drawing # 21668 a

[redacted]

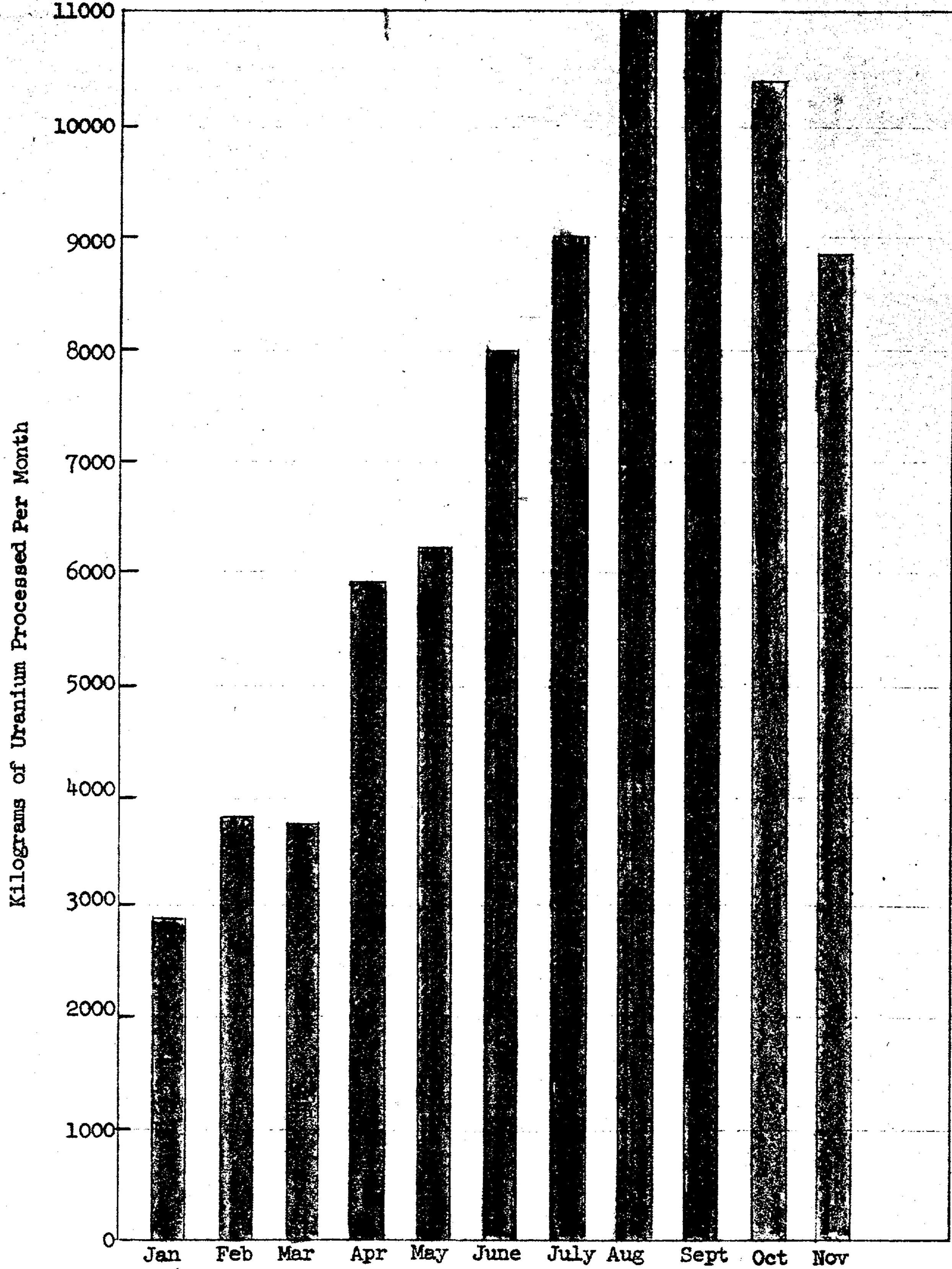

Figure 1.0

METAL RECOVERY PRODUCTION

2.0 PROCESS CHEMISTRY

2.1 Solvent Extraction Losses

2.11 Uranium Losses

Uranium losses to the IAW and ICW waste streams and to the plutonium (IBP) bearing stream are given in Table 2.11-1. These losses are based on flowing stream samples taken at 24 hour intervals during plant operation.

Table 2.11-1

Uranium Losses

Stream	Uranium Loss (% of IAF U)
Flowing stream	
IAW	0.06
IBP	< 0.01
ICW	0.12
Total	0.29
Composite	1.44

The composite uranium loss for the plant, including losses resulting from centrifuge cleanout and filtration operations plus solvent extraction loss, was 1.4 per cent.

2.12 Plutonium Losses

Listed in Table 2.12-1 are the plutonium losses to the first and second cycle waste streams and to the IBU stream.

Table 2.12-1

Plutonium Losses

Stream	Plutonium Losses (% of IAF Pu)
Flowing stream	
IAW	17.4
IBU	0.9
IIAW	0.1
IIBW	0.2
IIBPW	0.01
Total	18.6

The composite plutonium loss from the plant during the period that plutonium was being recovered was 30.5 per cent(1).

(1) Previous monthly reports have indicated that quantitative recovery of plutonium is not economically feasible.

2.2 Fission Product Decontamination

2.21 Uranium Decontamination

Through one cycle of operation, the fission product beta and gamma activities were separated from the uranium product by factors of 2.9×10^4 and 1.4×10^3, respectively.

Specific fission product decontamination factors of the uranium product from the feed are listed in Table 2.21-1.

Specific Decontamination Factors for Uranium Product

Constituent	D.F.
Gross β	3.7×10^3
Gross $\beta^{(a)}$	2.9×10^4
Gross γ	606
Gross $\gamma^{(a)}$	1.4×10^3
Ru β	772
Zr β	73
Nb β	260
TRE β	3.9×10^4

(a) Corrected for $UX_1 + UX_2$ activity.

2.22 Plutonium Decontamination

The gross beta and gross gamma decontamination factors for the plutonium product stream were 4.4×10^5 and 2.2×10^3, respectively.

2.3 Uranium Product Purity

The plutonium contamination in the uranium product averaged 27 ppb based on uranium. The fission product beta and gamma activities of the uranium product averaged 0.5 and 34 per cent, respectively, of old natural uranium.

The ionic impurities in the uranium product are listed in Table 2.3-1.

Table 2.3-1

Ionic Contamination in Uranium Product

Contaminant	Concentration PPM of U	Contaminant	Concentration
Al	2[b]	Mo	-
Ca	20	Ni	-
Cr	8	Pb	-
Cu	2[b]	Sn	-
Fe	900	PO_4	200
Mg	4[b]	Na	16[c]
		Si	19

(a) High iron contamination is due to entrainment of iron with the organic from the IB column.

(b) These values are the lower limit of analysis.

(c) By flame photometer.

3.0 PLANT OPERATION

The Metal Recovery plant operated 27 days to process 8,846 kg of uranium as 2 M uranyl nitrate hexahydrate solution. Approximately 42 grams of plutonium were recovered from the waste processed.

3.1 Limitations to Plant Capacity

During the latter part of the month the uranium content of the dissolved slurry dropped to 39.1 grams per liter, thereby decreasing the plant throughput by 10 per cent. In order to maintain a design capacity of 750 pounds of uranium per day it is necessary to have a dissolved slurry concentration of 43 g/liter.[1] Modifications to present recirculating methods and installation of an additional sparging port in tank W-10, failed to bring about an increased slurry concentration. An attempt will be made to increase the uranium concentration during December by "bleeding in" the scrap material obtained from Harshaw Chemical Works.

3.2 Feed Preparation

3.21 Slurry Dissolution

Approximately 49,489 gallons of metal waste slurry were pumped from waste tank W-10 to the Metal Recovery plant for feed make up. A volume of 8,140 gallons of concentrated nitric acid was added to the slurry for neutralization and uranium dissolution. The average analysis of the dissolved slurry was as follows:

Uranium	44.7 g/liter
Nitric Acid	1.38 M

3.22 Feed Evaporation

There was 55,629 gallons of dilute feed concentrated in the feed evaporator to obtain 23,463 gallons of concentrated feed containing 113 gm of uranium per liter and 3.3 moles of nitric acid per liter.

3.3 Solvent Extraction

Installation of Milton Roy pumps in place of rotameters has eliminated fluctuations in rate of solvent delivery caused by the build-up of crud on rotameter floats and walls.

Leakage rates on the column pulse pumps averaged 44 cc/mm for the month of November.

(1) CF 53-7-90

3.4 Plutonium Recovery

The average plutonium concentration in the metal waste solution was 6 gm of plutonium per ton of uranium. Approximately 42 grams of plutonium were recovered from the 66 grams obtained from the tank farm wastes.

3.5 Acid Consumption

During November, 8,900 gallons of concentrated nitric acid was consumed. An average of 12 moles of nitric acid was consumed per mole of uranium produced.

3.6 Solvent Recovery

The solvent raffinate streams from the IC column and the IIB column were contacted with 0.1 M Na_2CO_3 solution in an 8-inch pulse column. The usual water-washing of the solvent was eliminated during the month to allow removal of the 5-inch unpacked column.

3.61 Solvent Losses

Approximately 1,400 gallons of solvent were pumped to the columns per day with an average loss of 4 gallons per operating day. The overall solvent loss for the month was 0.3 per cent of the total solvent throughput.

3.62 Decontamination

Continuous scrubbing of the solvent raffinate streams with 0.1 M Na_2CO_3 effected a uranium decontamination factor of 8. Beta and gamma decontamination factors were negligible due to the insignificant amount of contamination in the solvent raffinate streams.

4.0 FEED MATERIAL

During November there were two shipments of irradiated slugs from Brookhaven National Laboratory containing 1,694 kg of uranium, and three shipments of waste material containing 5,667 kg of uranium in the form of sodium diuranate filter press cake from Harshaw Chemical Company.

5.0 SUMMARY OF PLANT OPERATING COST FOR OCTOBER

Due to the lag in the receipt of cost information, this section of the monthly report presents data for October rather than for the current month.

5.1 Gross Cost

The gross operating cost for the Metal Recovery plant for October was $44,092.

The component costs were:

Labor	$15,127
Material	11,338
Overhead*	17,627
Total	$44,092

* The term overhead includes expense allocation and other distributed charges.

5.2 Labor Cost Breakdown

The major labor charges for October were as follows:

	October
Pilot Plant Section	$8,642
Engineering and Maintenance Division	2,282
Analytical Chemistry	4,203
	$15,127

5.21 Analytical Services Labor Cost Summary

The Analytical Chemistry Division performed 1,560 analyses for the plant during October; the average cost per analysis was $5.01, including charges for labor, material, and overhead.

Laboratory	No. Analysis	Cost/Analyses*
C. E. Lamb	1300	$ 4.37
C. L. Burros	56	11.95
G. R. Wilson	62	4.34
C. Feldman	15	48.06
E. Wyatt	107	3.04
J. Cooper	20	7.49

* Includes charges for expense allocations.

5.22 Summary of Engineering and Maintenance Charges

During the month of October engineering and maintenance charges were $3,897. The labor charges amounted to $2,282 and the material charges were $1,615. Of the charges incurred only 33% (or $274 for material and $1,005 for labor) was due to Metal Recovery plant operation and maintenance. The remainder of the engineering and maintenance charges were for alterations and additions which are being made in preparation for the processing of the Hanford Metal wastes scheduled for February of 1954. Following is a summary of the October charges to the various work orders.

W. O.	Description	Material	Labor	Total
500283	Blanket covering repair and minor charges	$234.74	$730.00	$965.74
500375	Product shipping	40.24	274.00	314.24
2190	New 4-inch column	1,145.76	802.00	1,947.76
2193	Changes and Additions to Metal Recovery Plant to process Hanford Wastes	77.96	444.00	521.96
2195	Fabrication of three 7-inch Resin columns	116.30	31.00	147.30
		$1,615.00	$2,282.00	$3,897.00

5.3 Summary of Worked Material and Overhead Charges

A total worked material and overhead charge of $17,496 was assessed to the Metal Recovery plant during October. A detailed summary of each individual charge is as follows:

Description	Amount
Expense allocation	$8,989
Worked material	
Air supply	344
Electricity	802
S.F. material control	157
Steam	1,160
Tank farm	2,281
Treated water	204
Decontamination	262
Health Physics	1,918
Laboratory Research Directors Dept.	816
Chemical Technology Division Distributed	563
Total	$17,496

6.0 SAFETY

Each shift foreman conducted one safety meeting during November and all shift personnel attended the safety movie entitled "The Shocking Truth."

The average radiation exposure of personnel in the plant during November was 29 mr/week.

R. E. Brooksbank

REB:aeh